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Columbus, Ohio 10 April 1944 DSM a5-56p

NY. 48

MEMORANDUM FOR THE OFFICER IN CHARGE:

Subject : Contamination of Graphite.

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To : Interviews with V. C. Hammister and Dr. H. G. MacPherson.

On 7 April 1944 this officer interviewed V. C. Hammister Chief Chemist for the National Carbon Company and Dr. H. G. MacPherson, Research Physicist for the same company at their offices in the National Carbon Company Building, 1280 West 73rd Street, Cleveland, Ohio with reference to the contamination of Graphite produced by the National Carbon Company on contract with the Manhattan District. This investigation is based on a letter from the Chicago Branch Intelligence Officer and a request from the District Intelligence Officer.

The following is a composite of the information received from the above two men in the course of this interview:

The National Carbon Company has been engaged in the producing of graphite for the Manhattan District for several years. In November, representatives of the Manhattan District informed National Carbon Company that it was desired that a very pure form of graphite be produced. In accordance with this request the National Carbon Company began to produce a graphite known as the KC, KS, AGOT graphite. The initial K stands for the word Kendall Coke, the initial C stands for Chicago Pitch and the initial S stands for Standard Pitch. In other words the basic ingredients for the production of graphite are petroleum coke and pitch. Hence the KC graphite is produced from Kendall Petroleum Coke and Chicago Pitch and the KS graphite is produced from Kendall Petroleum Coke and Standard Pitch. The KC graphite is the product in which the contamination was first discovered and practically all data listed below refers to KC graphite.

The Kendall Coke used in all graphite involved in this investigation was produced by the Kendall Refining Company, Bradford, Pennsylvania, sold by the refining company to H. R. Deffler, Marine Bank Building, Buffalo, New York, broker in such products and sold by him to the National Carbon Company.

In order to produce Graphite of the purity necessary to meet the specifications of the Manhattan District it is necessary that the finished product have a boron content of not more than 5 parts per 10 million. The symbol used by the National Carbon Company to indicate the number of boron parts per 10 million is designated as 0#. Henceforth in this report that term will be used.

In producing the finished product seven different steps are involved. The entire process takes a period of two months if there is no time lost between each step. In event there is any time lost between steps then of course the procedure takes a longer time dependent upon the time lost. The steps in the procedure in chronological order are as follows:





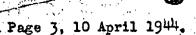
- 1. The Kendall Petroleum Coke is calcined. This is a heat treatment in which the petroleum coke is shrunk.
- 2. The coke is ground into a powder.
- 3. The powder is mixed with a pitch binder.
- 4. The resulting mixture is extruded through a dye and comes out in the form of a bar.
- 5. The bars are then placed in furnaces and surrounded with granular packing material and fired to a heat of 900 degrees Centigrade. This heat carbonizes or decomposes the pitch binder and converts it into carbon with the result that a porous carbon bar is produced which will not melt and which cannot be reduced to liquid.
- 6. The bars are placed in autoclave where they are impregnated with pitch under vacuum presure. The purpose of this is to fill the porous spaces with carbon to give a greater density.
- 7. The bars are stacked in a graphitizing furnace. Between these stacks of bars are placed petroleum coke. Heavyelectrical currents are then passed through the bars. When the bars come out of this furnace they constitute the finished product. These finished bars are 50 inches in length, 41 inches wide and 42 inches high.

Each graphitizing process is known as a "heat". Each heat represents 350 bars of a total weight of between 10 and 11 tons. From each heat a sample is taken of ten bars. These samples are tested to determine the 0# and the 0#'s of each sample are added together and an average taken to indicate the 0# of the entire heat.

The first 54 heats of KC graphite all indicated an 0# of five or less which meant that they all conformed to the specifications required. The next heat indicated an 0# of 6.5 but nothing was thought of this since the standard analysis deviation can be as high as 1.2. After this the next five heats were good. Following this the heats ran as follows:

Heat 61 - 0# 8-Heat 62 - 0# 9 Heat 63, 64, 65 - 0.K. Heat b6 - 0# 7 Heat 67 through 77 - 0.K. Heat 78 - 0# 6 Heat 79 - 0# 10 Heat 80 - 0# 6.5 Heat 81 - 0# 7 Heat 82 through 93 - 0.K. Heat 94 - 0# 20 Heat 95 - 0# 10 Heat 96 through 98 - 0.K. Heat 99 - 0# 84





Heat 100 - 0# 8

Heat 101 - 0# 8-

Heat 102 - 0# 17-

Heat 103 - 0# 7.5

· Heat 104 - 0# 7

(The plus and minus signs indicate that the boron content is just above or just below the number indicated.)

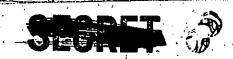
The next 17 heats following heat 104 all showed a high 0#, much above the amount allowed.

The management of National Carbon Company became concerned when the heats indicated above as heat No. 61 and 62 revealed an 0# of 8- and 9. The samples for these heats were shipped on February 18 and received in Cleveland for analysis on February 21. Steps were thereupon taken to determine the cause for the high O#. Samples were taken of the raw petroleum coke involved in the process. The samples revealed an O# of approximately three which would indicate that the raw petroleum coke was not the cause of the high 0#. In explanation it should be added that all raw petroleum coke contains boron in varying degrees. It was known also that samples had been taken of all lots of Chicago pitch immediately after it was distilled and the O# of these samples show 4 and 5 so that the original pitch could not be the cause of the high O#. In explanation of these samples it should be indicated that the samples of coke were taken from the original storage pile and that the samples of the pitch were made at the point of origin so that it was still possible for these products to have acquired additional boron by some means or other during shipment to Clarksburg. West Virginia or in the plant before they were actually used in the process of producing the finished graphite. The pitch was to be shipped from Chicago and the coke from Welland, Ontario...

Investigation was then made by Mr. Hammister to determine other possible causes of contamination. It was discovered that welding work had been done on the railing surrounding the impregnator involved in step No. 6 of the process and that below the railing Were open containers of pitch. Tests of the flux used in welding revealed that it contained 58% boron. It was thought then that some of this flux had dropped into the drums of pitch and caused the high boron content. However if this had been the case each succeeding heat after the two bad heats mentioned above would have shown a gradual tapering off in the amount of boron. However the heat immediately after the two bad heats was a good one. Hence it was decided that the welding process was not the cause.

Mr. Hammister next investigated the raw piles of petroleum coke. All of the petroleum coke involved in producing the KC graphite was located in storage piles in Welland Ontario. The calcining process was also carried on at Welland, Ontario and the resulting product then shipped to the Clarksburg, West Virginia plant of National Carbon Company for further processing. Mr. Hammister discovered that originally all of the petroleum coke was stored in a pile known as pile No. 28. For purposes of accounting and inventory it was decided to create two new piles known as piles 8 and 25. The coke contained in pile 28 was first used up and this was the coke used in the original heats. After this coke was used up coke was taken from the new piles known as 8 and 25. Investigation revealed that piles 8 and 25 were located on land which had been freshly filled and that the material used for filling this land was furnace slag. Mr. Hammister took a sample of this





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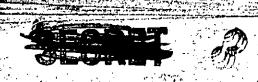
Furnace slag bottom, tested it and found that it had an O# of 400. The material from these piles is scooped up by a large mechanical shovel. Furthermore Mr. Hammister stated that these piles have been completely depleted and scraped completely clean. His feeling now is that in scooping up the petroleum coke a sufficient amount of the slag was also scooped up to account for the high O# found in the various heats. This would also in Mr. Hammister's opinion account for the spottiness of the high O# in the various heats sampled since it would be logical to assume that some portions of the coke would not contain the slag while others would.

After a number of bad heats were discovered samples were taken of other individual bars contained in these heats and one bar was found to have an 0# of 43. In order for an 0# of 43 to be produced by the bottom material or slag it would be necessary that 10% of the bottom material show an 0# of 400. This is one reason why additional samples are being taken in order to determine whether the slag universally has an 0# of 400 or better. Mr. Hammister and Dr. MacPherson estimated that it would take about five ton of slag having an 0# of 400 to produce the composit which has been found. This five tons would actually constitute .2% of the total tonnage of pile 8 and 25. Mr. Hammister has requested that ten more samples of the bottom material under pile 8 and 25 be forwarded to him for analysis in order that this theory can be more fully confirmed. These samples were shipped on April 4 and the results of the analysis of these samples should be available by April 14 or 15.

So far as introduction of material containing boron by a saboteur is concerned Mr. Hammister and Dr. MacPherson feel that the time to do this would be in the first three processes when the material is still in a powder form. Probably process No. 3 which is the mixing process would be the best. The material is in an open container before it is introduced into the mixer. Mr. Hammister said that 6.8 grams of boric acid added to each mix of 650 pounds would produce the contamination found. In other words a teaspoon full thrown into each mix would contain the contamination. . According to Mr. Hammister approximately 50 employees would have an opportunity to contaminate the material during the first three processes. Mr. Hammister stated that there is no reason to suspect any of the employees and there has been nothing to indicate any suspicions. In addition he stated that there are only six employees at the National Carbon Company plant at Clarksburg, West Virginia who know that boron would contaminate the graphite. These men are key employees and highly trusted. In other words this is a closely guarded secret. The average employee would not know anything about the contaminating feature of boron. In other words according to Mr. Hemmister either an enemy agent would have had to be introduced into the plant or one of the employees bought Off by an enemy agent. Mr. Hammister also added that the operations employees do not know the purpose for which the graphite is being made and do not know the destination of the finished product. Only the six key men previously mentioned know the destination of the product and these men know nothing more than that.

All of the KC product has now been completed and all of the KS product under the present order has passed through the first five stages of the process and it is not contemplated that any more of the KC or KS graphite will be produced unless a new order is received. Hence in event the high O# has been caused by sabotage there is little chance of any further sabotage of this type in as much as it is believed that sabotage through introduction of boron would have to occur during the





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first three steps of the process. Since the high O# has been discovered all graphitizing (this is No. 7 stage in production) has been stopped. The National Carbon Company is in the process of checking the sixteen thousand bars which are now awaiting graphitizing. A composite sample is being taken of these bars amounting to about 2% of all bars to determine the O# of these bars. These sixteen thousand bars are all KS bars. Six thousand of these bars are located at Morganton, North Carolina where part of the graphitizing work is done and ten thousand are located at Clarksburg, West Virginia.

The only other explanation for the high 0# of the bars is that the material was shipped in railroad cars in which boron or some product containing a high boron content has previously been shipped and that residue from such shipment had mixed with the material. There is no indication that this has occurred and it would be practically an impossibility to check this theory.

Arrangements were made with Mr. Hammister to notify this officer as soon as the results of the analysis of the samples of furnace slag have been determined. This information will be forwarded immediately to District Headquarters.

JOHN L. DAVIES, JR.

Columbus Branch Intelligence Officer.

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